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A METHOD FOR POSITIVE COLLECTION OF IODINE FROM AIRBORNE CARBON-IODINE BONDED COMPOUNDS

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ABSTRACT

A method for quantitative collection of iodine from carbon-iodine bonded compounds is described. This method utilizes a high frequency electric spark to rupture the carbon-iodine bond followed by collection of the resultant ionic and molecular iodine forms on ion-exchange resin.

PREFACE

This method was developed by the authors for the Bioenvironmental Research Program, Southwestern Radiological Health Laboratory, as a part of the Iodine Investigation Studies they are conducting under the sponsorship of the Atomic Energy Commission.

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Introduction

This paper is published as part of a comprehensive study toward development of an efficient sampling system for all forms of airborne iodine.

Recent evidence has indicated that the iodine from gaseous carbon-iodine bonded compounds is not efficiently collected by air sampling systems which use either activated charcoal[1,2] or basic ion-exchange resin[3] as adsorbents. As it is known that both activated charcoal[4,5,6] and basic ion-exchange resin[7] display excellent retentive properties for gaseous ionic and molecular forms of iodine, a method for practical immediate rupture of the carbon-iodine bond would prove meaningful.

Spark discharge was preferred for such dissociation both from kinetic and thermochemical considerations. The stoichiometry[8] of the pyrolysis should follow the equation:

$$2RI \rightarrow \text{olefin} + RH + I_2$$

with some minor contribution from

$$RI \rightarrow \text{olefin} + HI$$

Unsaturated or conjugated iodides such as allyl I or $\phi CH_2 I$ [9] follow a simpler course:

$$2RI \rightarrow R_2 + I_2$$

This is also true of di-iodides[10,11] which proceed as

$$RI_2 \rightleftarrows \text{hydrocarbon} + I_2$$

Thus in every case, the pyrolysis of carbon-iodine bonded compounds leaves the iodine in either the molecular or the iodide form.

Materials and Methods

The apparatus (Figure 1) used for determining the efficiency of spark discharge for carbon-iodine bond rupture consisted of: a flow-through type glass generator flask, a dual tungsten electrode glass reaction chamber using a Tesla coil as spark source, dry, strongly basic ion-exchange resin (Dowex 1 X-8, 50-100 mesh) packed in a 7.6 cm by 3.2 cm I.D. (3"x 1.25" I.D.) plexiglass cylinder fitted at either end with Millipore filters (MF Type HA, 0.45μ $\pm$ 0.02μ), and a Gelman pump (Model 13152) and rotameter.

Basic ion-exchange resin was used in preference to charcoal for adsorption of inorganic ionic and molecular iodine forms because of its stronger iodine bond and its lower rate of surface erosion. Surface erosion causes iodine losses due to particulate escape through the Millipore filter, i.e., particles smaller than 0.45μ.

All recoveries were determined at room temperature (25 to 30°C) with the air flow regulated between 14.1 to 19.8 lpm (0.5 to 0.7 cfm) during and for five additional hours after volatilization. The compounds tested were labeled methyl, ethyl, butyl, octyl, and phenyl iodides. These compounds were labeled by 14 Mev neutron activation utilizing the (n, 2n) reaction. A Texas Nuclear (Model 9500) generator provided the neutron source. This method of labeling provides rather low specific activity iodine compounds. The volume of the compounds used in the experiments ranged from 0.1 to 1.0 ml.

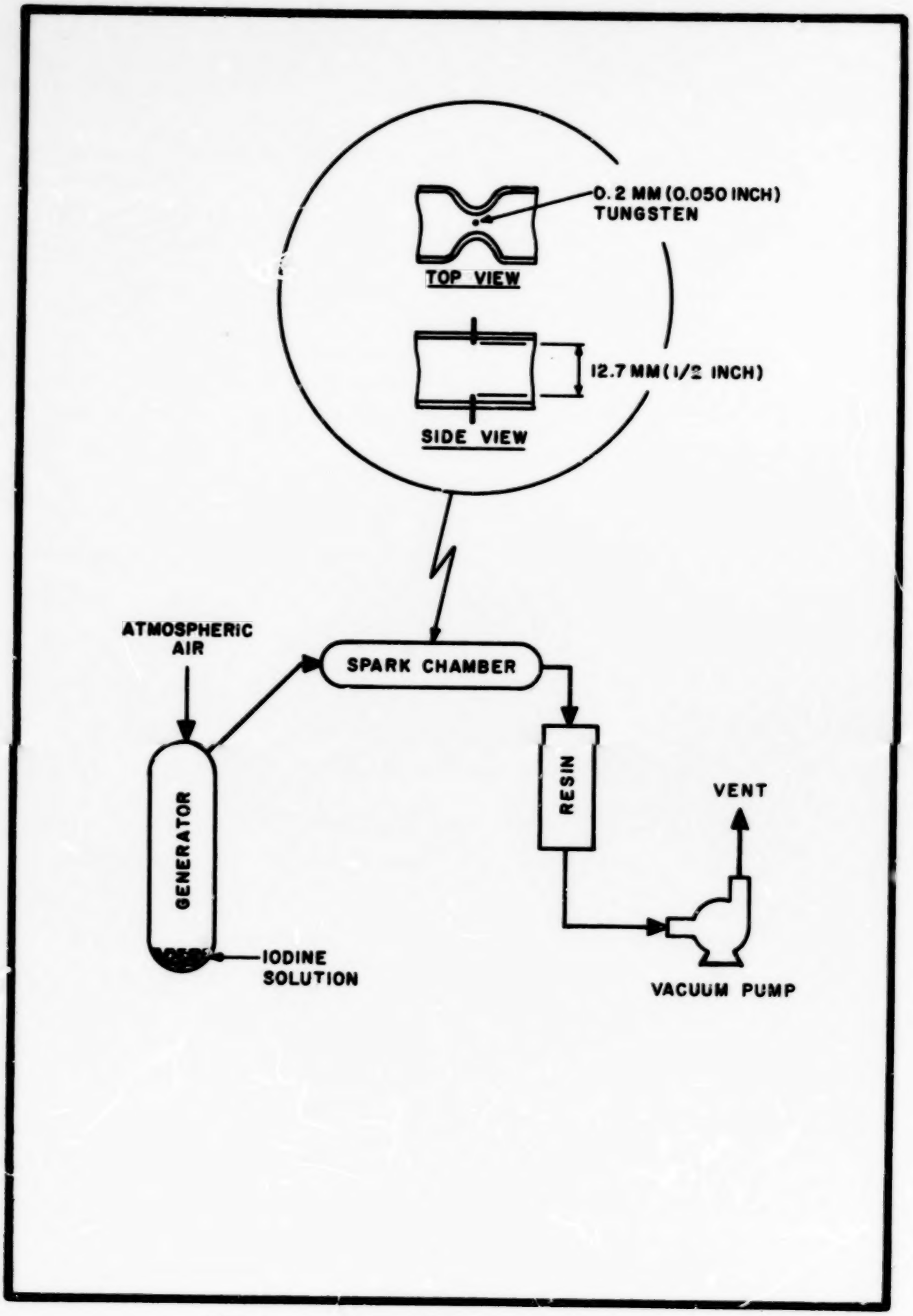

Figure 1. Flow diagram of apparatus.

Results and Discussion

When the spark was not used during volatilization, very low retention was exhibited by basic ion-exchange resin for iodine in the above compounds (Table 1). However, when the spark discharge was used during volatilization, quantitative retention of the iodine was displayed by the resin for all the above compounds (Table 2). The results are even more striking considering the low specific activity of the labeled iodine compounds. Thus the iodine in carbon-iodine bonded compounds can be permanently and quantitatively collected by suitable ionic and molecular iodine absorbers by first dissociating the compounds by spark discharge.

Table 1. Iodine recoveries without using spark discharge.

Compound Tested	Activity Added (cpm)	Activity Recovered (cpm)
Methyl Iodide	28,755	2,444
Ethyl Iodide	8,137	1,196
Butyl Iodide	12,276	1,079
Octyl Iodide	22,473	3,724
Phenyl Iodide	9,276	925

Table 2. Iodine recoveries using spark discharge.

Compound Tested	Activity Added (cpm)	Activity Recovered (cpm)
Methyl Iodide	14,264	15,102
Ethyl Iodide	6,274	6,241
Butyl Iodide	8,991	8,576
Octyl Iodide	12,843	11,988
Phenyl Iodide	7,745	7,700

A sampler for airborne iodine, which incorporates a spark discharge to dissociate any carbon-iodine bonded compounds, has been fabricated. Tests are currently under way for development of pertinent parameters, i.e., discharge energy, discharge frequency, coefficients of energy absorption of the respective gasses, and adsorption bed thickness.

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